AFATL-TR-82-37

Ballistic Irregularities of Triaminoguanidine Nitrate (TAGN) Propellants

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BALLISTICS BRANCH
DIRECT FIRE WEAPONS DIVISION

MAY 1982

FINAL REPORT FOR PERIOD FEBRUARY 1980-DECEMBER 1981

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Air Force Armament Laboratory

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REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER AFATL-TR-82-37	2. GOVT ACCESSION NO. AD-A120 347	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) BALLISTIC IRREGULARITIES OF TRIAMINOGUANIDINE NITRATE (TAGN) PROPELLANTS		5. TYPE OF REPORT & PERIOD COVERED Final Report: February 1980-December 1981
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Bertram K. Moy		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS Direct Fire Weapons Division Air Force Armament Laboratory Eglin Air Force Base, Florida 32542		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS PE: 62602F JON: 2560-08-20
11. CONTROLLING OFFICE NAME AND ADDRESS Air Force Armament Laboratory Armament Division Eglin Air Force Base, Florida 32542		12. REPORT DATE May 1982
		13. NUMBER OF PAGES 36
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

Availability of this report is specified on verso of front cover.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

TAGN
TAGN Propellants
TAGN Impurities

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Burning rates from propellants containing Triaminoguanidine Nitrate (TAGN) were obtained at pressures between 2,000 and 50,000 psi. Impurities in the TAGN and the isopropanol (IPA) desensitizing diluent generally caused microporosity and erratic burning rates in propellants. Most of these impurities could be removed by washing the TAGN with a polar solvent in which they were soluble. Propellant made with this TAGN had improved ballistic properties. Storage of recrystallized TAGN in IPA resulted in impurity formation. Propellants made

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20. ABSTRACT (CONCLUDED)

with TAGN washed or ground in Freon® 113 exhibited erratic burning rates. Drying of TAGN from IPA in a high humidity atmosphere resulted in TAGN agglomerates. Propellants made with these agglomerates exhibited localized discolorations, voids, and erratic burning rates. The study implied that propellants made with TAGN containing minimal impurities should give good ballistic data. TAGN which contains impurities can usually be treated to yield a TAGN which, when incorporated into a propellant, gives a good burning rate.

PREFACE

This report documents work performed at the Air Force Armament Laboratory, Armament Division, Eglin Air Force Base, Florida, between February 1980 and December 1981 in support of Project 25600820. Mr. Bertram K. Moy (DLDL) was Project Engineer for this effort.

The Public Affairs Office has reviewed this report, and it is releasable to the National Technical Information Service (NTIS), where it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication.

FOR THE COMMANDER

MARVIN J. WOODRING, Colonel, USAF
Chief, Direct Fire Weapons Division

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SECTION I

INTRODUCTION

Triaminoguanidine Nitrate (TAGN) is an essential ingredient used in an alternate propellant developed for the GAU-8 gun. It contributes to lower molecular weight gases and reduced flame temperature concomitant with increased impetus and muzzle velocity. The appearance of colored impurities in TAGN delivered to Eglin Air Force Base, Florida, provoked concern over the ballistic and stability characteristics of propellants made with this TAGN. Work by Hartman, et al., (Reference 1) indicated that small amounts of colored impurities did not affect gun propellant stability. Work by Robb, et al., (Reference 2) indicated that results from safety tests on impure TAGN did not significantly differ from those on pure TAGN. Fong (Reference 3) indicated that burning rate data generated at Eglin AFB had shown ballistic irregularity which could be traced to TAGN impurities. It is important to propellant manufacturers to know how the impurities in TAGN will affect the ballistic properties of propellants. This report documents the burning rate data generated at Eglin AFB on propellants made from various lots of TAGN.

SECTION II

TECHNICAL DISCUSSION

BACKGROUND

The problem associated with impurities in TAGN did not receive wide attention until a large shipment of TAGN desensitized with isopropanol (IPA) was delivered to Eglin AFB, Florida, from Rocketdyne in 1978. Previously, TAGN was generally handled as a dry material or, if wet with a diluent, was used within a few days. Examination of some of the TAGN shipment revealed that the IPA diluent exhibited various shades of yellow, brown, and pink. The crystalline TAGN was generally white, although a few containers had TAGN with a pinkish tinge. Considerable effort was expended at Eglin AFB and other laboratories to isolate and identify the impurities in the TAGN and IPA with little or no success (References 1 and 2). Although these laboratories indicated that the impurities did not affect the sensitivity or stability of TAGN or TAGN gun propellants, preliminary data indicated that propellant grains prepared to a standard formulation exhibited higher gun pressure when manufactured from impure TAGN, indicating the presence of microporosity. Density measurements of gun propellants made with TAGN prepared from cyanamide, Equation (1), were consistently lower (Reference 4) than the density measurements of propellants made with TAGN prepared from guanidine nitrate, Equation (2).

$$\underset{\text{Cyanamide}}{H_2N\text{-}C{\equiv}N} + HNO_3 + 3H_2N\text{-}NH_2 \xrightarrow[\Delta]{H_2O} \underset{\text{TAGN}}{\left(H_2N\text{-}\overset{H}{N}\right)_2 C{=}N\text{-}NH_2 \cdot HNO_3} + 2NH_3 \quad (1)$$

$$\underset{\text{Guanidine Nitrate}}{H_2N\text{-}\overset{NH}{\overset{\|}{C}}\text{-}NH_2 \cdot HNO_3} + HNO_3 + 3H_2N\text{-}NH_2 \xrightarrow[\Delta]{H_2O/IPA} \underset{\text{TAGN}}{(H_2N\text{-}\overset{H}{N})_2 C{=}N\text{-}NH_2 \cdot HNO_3} + 3NH_3 \quad (2)$$

As mentioned above, these higher gun pressures were presumed to be a function of microporosity in the propellant grains. The apparent higher burning rates in gun firings prompted a study at Eglin AFB to determine strand burning rates of propellants made with TAGN from different lots and which had undergone different treatments.

DISCUSSION

A simple formulation (Table 1) was selected for the ballistic evaluation of TAGN.

TABLE 1. FORMULATION FOR BALLISTIC EVALUATION OF TAGN

INGREDIENT	WEIGHT PERCENT
Nitrocellulose (12.6N)	20.0
Resorcinol	0.2
Dibutyl Phthalate (DBP)	4.8
TAGN	75.0

Six lots of TAGN, listed below, were arbitrarily selected for evaluation.

LOT NO.	COLOR OF IPA DILUENT	COLOR OF TAGN
R-1	Dark Yellow	White
2-7-1	Very Light Yellow	White
2-7-5	Light Pink	Light Pink
1974	Brown	White
B-S	Dark Yellow	White
6229	No Visible Liquid	Light Yellow

Lots R-1, 2-7-1, 2-7-5, and Batch 5 were made from cyanamide, nitric acid, and hydrazine at Rocketdyne. Lots 1974 and 6229 were made from guanidine nitrate and hydrazine at Rocketdyne and Allegany Ballistics Laboratory, respectively. The propellants were processed in ethyl acetate (ETAC) and ethyl alcohol (ETOH), extruded into 1/8- to 1/4-inch diameter burning rate strands and tested at pressures between 2,000 and 50,000 pounds per square inch (psi) in the high pressure strand burner at Eglin AFB. The exact treatment of the TAGN prior to incorporation into the propellant is described in Appendix A. The range of burning rates of this formulation with ground TAGN is depicted in Figure 1. The nominal burning rate of this formulation is shown as the dotted line and appears in the other figures for reference. The burning rates in Figures 12 through 15 lie off the reference line because these batches were made with unground TAGN and had different ballistic characteristics.

Two batches of propellant strands were prepared with TAGN taken directly from the shipping containers, filtered (without washing), and ground* in ETAC (10 to 15μ). As can be seen in Figure 2, the strand burning rates from GP 169 (TAGN Lot R-1) were quite reproducible throughout the pressure range, while the burning rates (Figure 3) from GP 172 (TAGN Lot 1974) were very erratic and

*Grinding was accomplished in a 2.6-gallon SWECO Vibro-Energy Mill.

nonreproducible. It appeared that the impurities in TAGN Lots R-1 and 1974 were either different and/or had different effects on the propellants and their ballistic properties. Some of the TAGN Lot 1974 was recrystallized to remove impurities caused by the IPA diluent, or metallic ions in the IPA, and any occluded impurities in the TAGN crystals. This TAGN was subsequently ground in ETAC and arbitrarily named R-4. The burning rates (Figure 4) from GP 200 made with TAGN (R-4) were quite reproducible. Comparison of the data between Figures 3 and 4 indicated that the impurities in TAGN 1974 had a dramatic influence on propellant burning rates and that an improvement in ballistic properties could be achieved by removing these impurities.

When TAGN Lot 1974 was recrystallized and ground (R-4), the IPA diluent in which it was stored was colorless. After several months of storage, the IPA diluent became brown in color. This indicated that twice recrystallized, ground TAGN, with minimal occluded impurities, could continue to dissociate to Triaminoguanidine (TAG) in IPA to yield colored impurities caused by the IPA or by the metal ions in the IPA. Two propellant batches were made with this TAGN washed with IPA (GP 218) and IPA/hexane, hexane (GP 219). Chang, et al., (Reference 5) had indicated that removal of the color in the diluent was dependent upon the solubility of the impurity in the wash solution. After three washings with IPA, the IPA appeared colorless again. GP 219 was made with TAGN which was washed with an IPA/hexane mixture to remove the colored impurities and then washed in hexane. Hexane was used for the final wash to prevent reformation of impurities, since Hartman, et al., (Reference 1) had indicated that colored impurities form extremely slowly in non-polar liquids because of reduced solubility of the TAGN. Comparison of the burning rate data (Figures 4, 5, and 6) indicated that reproducible burning rates could be obtained from ground TAGN which had the impurities removed by washing and that the burning rates of propellant made with washed TAGN were essentially the same as those of propellant made with TAGN prior to impurity formation. This ballistic improvement implied that these impurities were oxidation products on the surface of the TAGN which were soluble in IPA.

Extremely erratic burning rate data were obtained for propellants made with TAGN which had been treated with Freon®. GP 228 was made with TAGN Lot B-5 which was ground in ETAC, washed with IPA/hexane, hexane, and then with Freon® 113 to prevent TAGN agglomeration. The burning rates from this batch (Figure 7) were erratic and nonreproducible. Some of the TAGN Lot 2-7-1 was ground in Freon® 113 in an attempt to reduce the possibility of impurity formation during grinding.

Hartman, et al., (Reference 1) had concluded that TAGN impurity formation in Freon® was unlikely because of TAGN insolubility. The burning rate data (Figure 8) from GP 234 made with this TAGN were the most erratic ever seen and showed no correlation with increasing pressure. A gas chromatographic analysis indicated no residual Freon® in the TAGN. Three propellant batches were made with this TAGN which was given additional treatments to determine if this erratic ballistic behavior could be improved. GP 246 was made with this TAGN which was redried in the oven with periodic agitation for exposure of additional surface area to air. The data (Figure 9) indicated that this treatment of the TAGN was successful since the burning rates fell back into the customary range with a minor degree of scatter. The IPA wash, which had previously been shown to be effective in removing impurities, and a methylene chloride (CH_2Cl_2) wash both proved to be ineffective in improving the ballistic characteristics of propellants made with this TAGN. Both GP 247 (IPA wash, Figure 10) and GP 248 (CH_2Cl_2 wash, Figure 11) exhibited erratic burning rates, although the rates from GP 248 were less erratic. The results from this series of batches implied that Freon® 113 or metal ions in Freon® somehow contributed to ballistic irregularity in the propellant, possibly through reaction with TAGN to form an impurity. This impurity was not removed through washing with IPA or CH_2Cl_2, but it could be oxidized to a nonreactive species.

A number of propellant batches were made with unground TAGN from Lot 2-7-1. This particular lot of TAGN only had a tinge of yellow in the IPA with no discoloration of the crystalline TAGN. Because of the minimal indications of impurities, it was expected that a propellant made with this TAGN would exhibit good ballistic characteristics. GP 215 was made with TAGN (Lot 2-7-1) removed from the storage container and dried without any washing. During the drying cycle at 48°C, the propellant strands changed from a cream color to a deep blue, with wet spots on the surface that did not ever dry. The copper screen on which the strands were drying became green in color. Analysis of the propellant indicated a highly acidic condition (pH 2.8). Apparently under these conditions, the following dissociation had taken place:

$$\left[C\left(\overset{H}{N}\text{-}NH_2\right)_3\right]^+ NO_3^- \longleftrightarrow \left(H_2N\text{-}\overset{H}{N}\right)_2 C{=}N\text{-}NH_2 + HNO_3$$

Fong (Reference 3) and Chang, et al., (Reference 6) had speculated that air oxidation of TAG could result in intensely colored Azo compounds, i.e.:

$$2\ \mathrm{TAG} \xrightarrow{O_2} \begin{matrix} H_2N\text{-}NH \\ H_2N\text{-}NH \end{matrix}\!\!>\!C{=}N\text{-}N{=}N\text{-}N{=}C\!<\!\!\begin{matrix} NH\text{-}NH_2 \\ NH\text{-}NH_2 \end{matrix} + 2H_2O$$

The burning rates from GP 215 (Figure 12) were erratic and nonreproducible, especially above 10,000 psi. The next three batches of propellant were made with this TAGN with additional treatment to determine if the propellant ballistic characteristics could be improved by changing or removing the impurities. Comparison of the burning rate data (Figures 12, 13, 14, and 15) indicated that air oxidation (by agitation of TAGN during drying), IPA and hexane washing, and IPA washing of TAGN Lot 2-7-1 all contributed to a marked improvement in the reproducibility of propellant burning rates. However, these treatments were not completely effective; some data scatter was still apparent. This could be attributed to the residual impurities occluded in the unground TAGN crystals and/or incomplete removal of impurities which could cause microporosity in the propellant.

Three additional batches of propellant were made to evaluate the burning rates of the following types of TAGN: (1) Lot 2-7-1, doubly recrystallized and ground, (2) Lot 6229, manufactured from guanidine nitrate and ground, and (3) Lot 2-7-5, manufactured from cyanamide and ground. All three propellants (CF-1, CF-2, and CF-3) exhibited erratic burning rates (Figures 16, 17, and 18) and contained black spots and purplish-black voids. The purplish-black holes were attributed to local concentrations of IPA in TAGN agglomerates formed during the drying of the TAGN in a high humidity environment. A high humidity situation exists at Eglin AFB at least eight months out of the year. During the propellant drying cycle, air oxidation of solvents or impurities in the solvents could produce relatively high concentrations of either free HNO_3 (or gases derived from reactions of HNO_3) or N_2 gas from thermolysis of oxidation products (Reference 3). The effects of the impurities and voids in the propellant were the dominant factors and overshadowed the effects of the different types of TAGN being evaluated.

Most of the data generated in this study came from propellant made with TAGN ground and dried from a diluent. On a larger manufacturing level, the TAGN is dried directly from the storage containers and ground by a fluid energy mill process. Trace metals may remain in the TAGN since it does not receive additional washing. Reactions of dissolved TAGN on the propellant surface with the trace metals could result in impurity formation. A NC/TAGN/RDX propellant sample made with impure TAGN at the Defence Research Centre, South Australia, and stored in a high humidity atmosphere, became yellow on all exposed surfaces. Stability and ballistic data should be generated for propellants made with TAGN under high humidity conditions.

This study has demonstrated that good propellant burning rate data can be obtained with TAGN containing minimal impurities. It was also shown that TAGN which contains impurities can generally be treated by washing, recrystallization, and/or air oxidation to yield a TAGN which, when incorporated into a propellant, gives good ballistic properties. Hartman, et al., (Reference 1) and Robb, et al., (Reference 2) have shown that the stability and sensitivity of propellant made with impure TAGN were adequate. These two studies, in conjunction with this study, indicated that propellants made with TAGN with minimal impurities should have good ballistic, sensitivity, and stability characteristics.

SECTION III
SUMMARY AND CONCLUSIONS

SUMMARY

This report documents the work done at Eglin AFB on the burning rates of propellant made from different lots of TAGN. The appearance of colored impurities in the IPA diluent for TAGN should be a cause for concern since these impurities could be incorporated into the propellant. Generally, these impurities caused microporosity in the propellants and erratic burning rates. The TAGN used in propellants which gave erratic burning rate data was recrystallized and incorporated into propellants which gave reproducible burning rate data. Even doubly recrystallized TAGN formed impurities after storage in IPA. The colored impurities in IPA diluent in ground TAGN could be washed out with IPA, and propellants made with this washed TAGN gave reproducible burning rates. Colored impurities in TAGN which had been dried were air oxidized through exposure of additional surface area into nonreactive species which had no apparent deleterious effect on propellant burning rates. Propellant made with TAGN which had been washed or ground in Freon® exhibited erratic burning behavior. Although washing of this TAGN with IPA or CH_2Cl_2 did not remove the suspected impurity, air oxidation was effective in producing a nonreactive species which did not contribute to propellant microporosity. The existence of impurities in IPA which was colorless was demonstrated in propellants made with Lot 2-7-1, where the propellant itself turned deep blue. Except for the effect of occluded impurities, washing with IPA or air oxidation was an effective method for negating the impurity effect on the propellant. Drying of TAGN from IPA in a high humidity atmosphere resulted in TAGN agglomeration. This resulted in high concentrations of IPA which were exhibited as dark spots or holes in the propellant, either or both of which contributed to ballistic irregularity.

CONCLUSIONS

1. TAGN should not be stored in IPA or any diluent in which TAGN has some solubility because of impurity formation.

2. Impurities in solution can generally be removed by washing with IPA and presumably with other polar solvents. Physically entrapped impurities can be removed by washing the TAGN after grinding.

3. Impurities in dried TAGN can be generally air oxidized to nonreactive species.

4. Impurities in TAGN can contribute to microporosity and erratic burning rates in propellants.

5. TAGN should not be treated or stored in Freon® because of impurity formation. Propellants made with this TAGN exhibited erratic burning rates.

6. Impurities in TAGN caused by Freon® treatment were not removed by washing with IPA or CH_2Cl_2.

7. Drying of IPA diluent of TAGN in a high humidity atmosphere could result in entrapped IPA in TAGN agglomerates. Propellants made with these agglomerates exhibit localized discoloration, voids, and erratic burning rates.

8. Grinding the TAGN in a fluid energy mill should resolve some of the above problems, i.e., (a) entrapped impurities will be released, (b) impurities can be oxidized to nonreactive species, and (c) TAGN agglomeration during drying and solvent entrapment in the agglomerates will be minimized.

9. Propellant made with TAGN containing minimal impurities should have good ballistic properties.

10. TAGN containing impurities can generally be treated to yield a TAGN which, when incorporated into a propellant, gives good ballistic properties.

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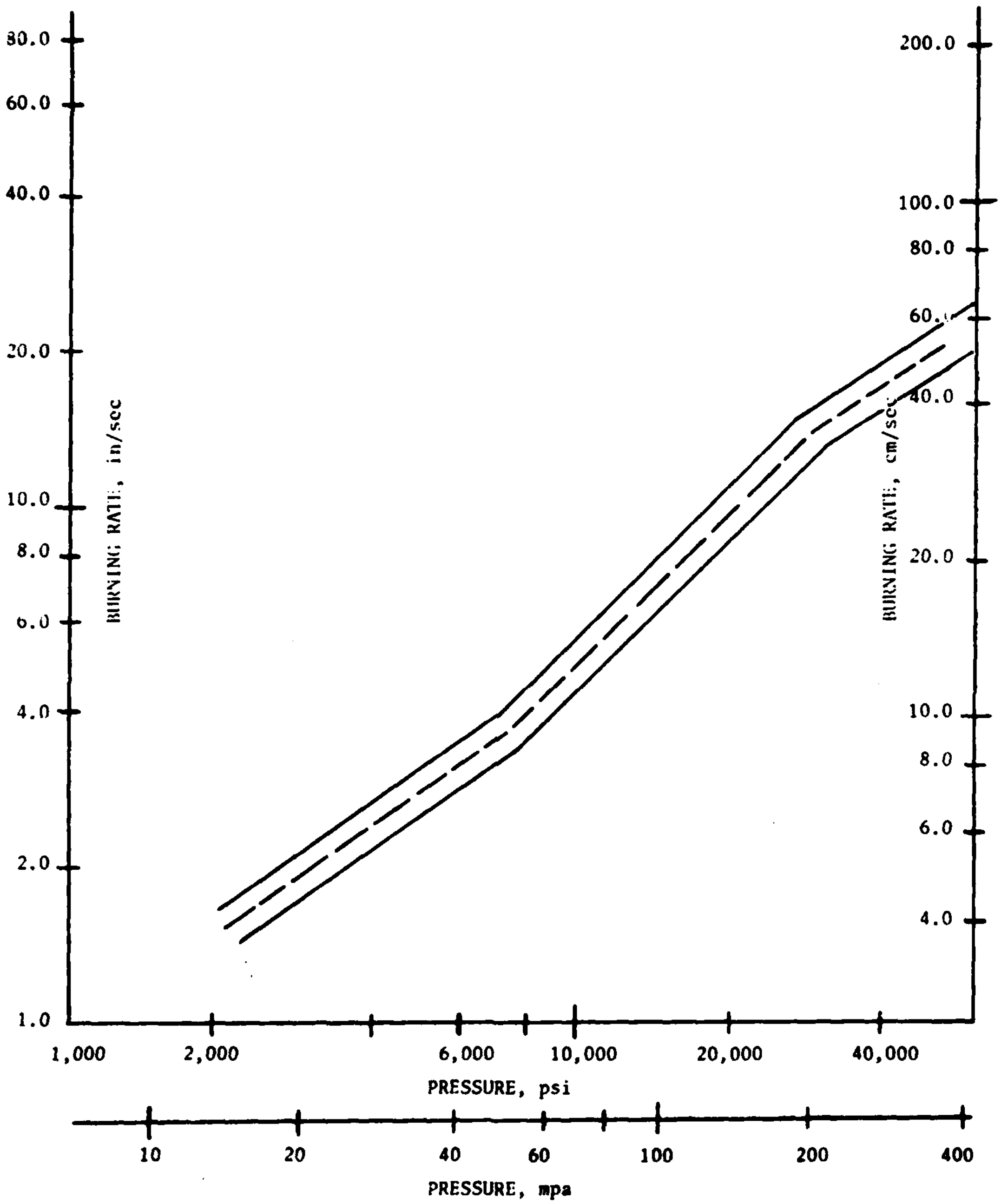

Figure 1. Nominal Burning Rates of a TAGN Propellant (NC/DBP/TAGN) (20/5/75)

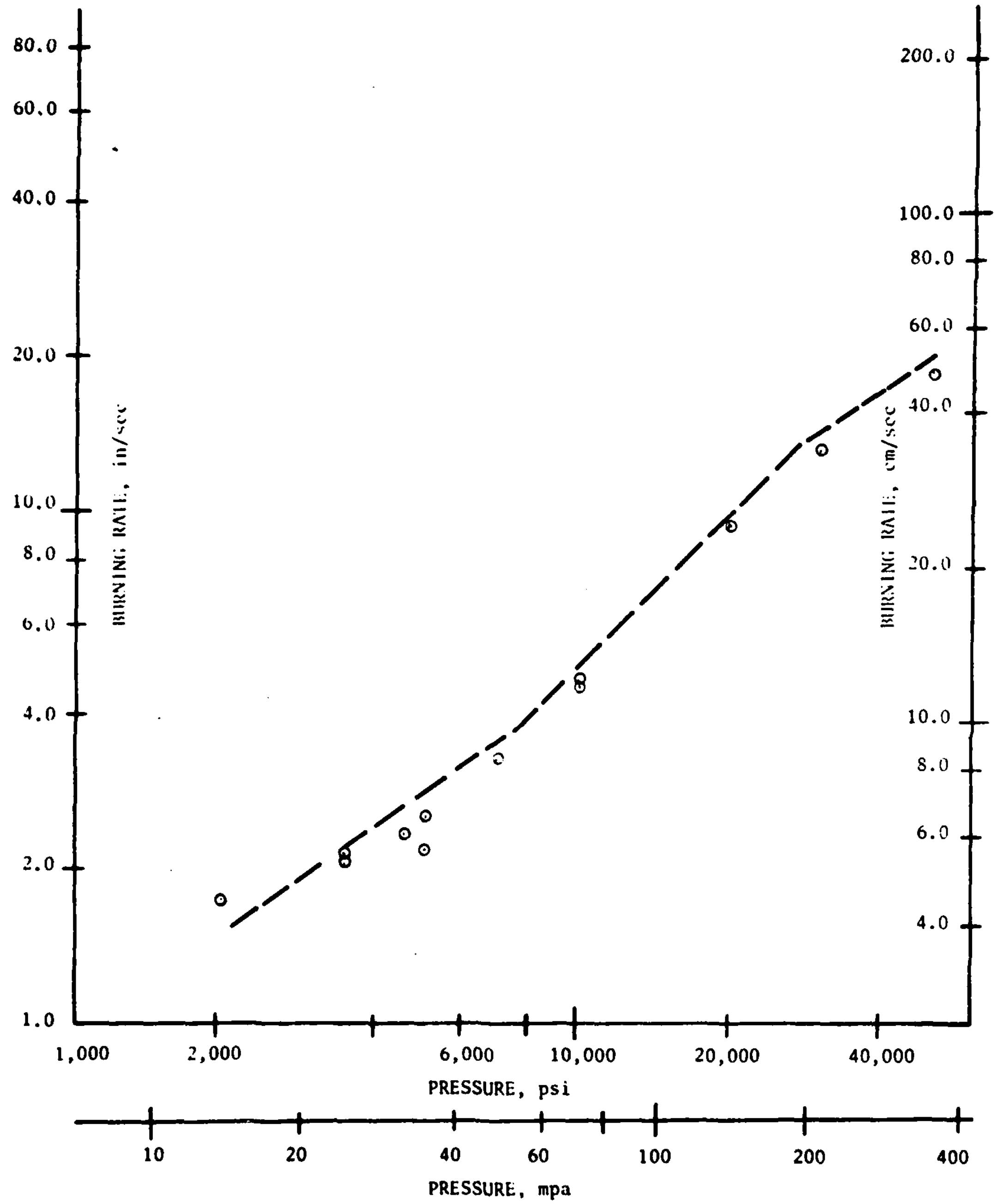

Figure 2. GP 169 Lot R-1, Ground

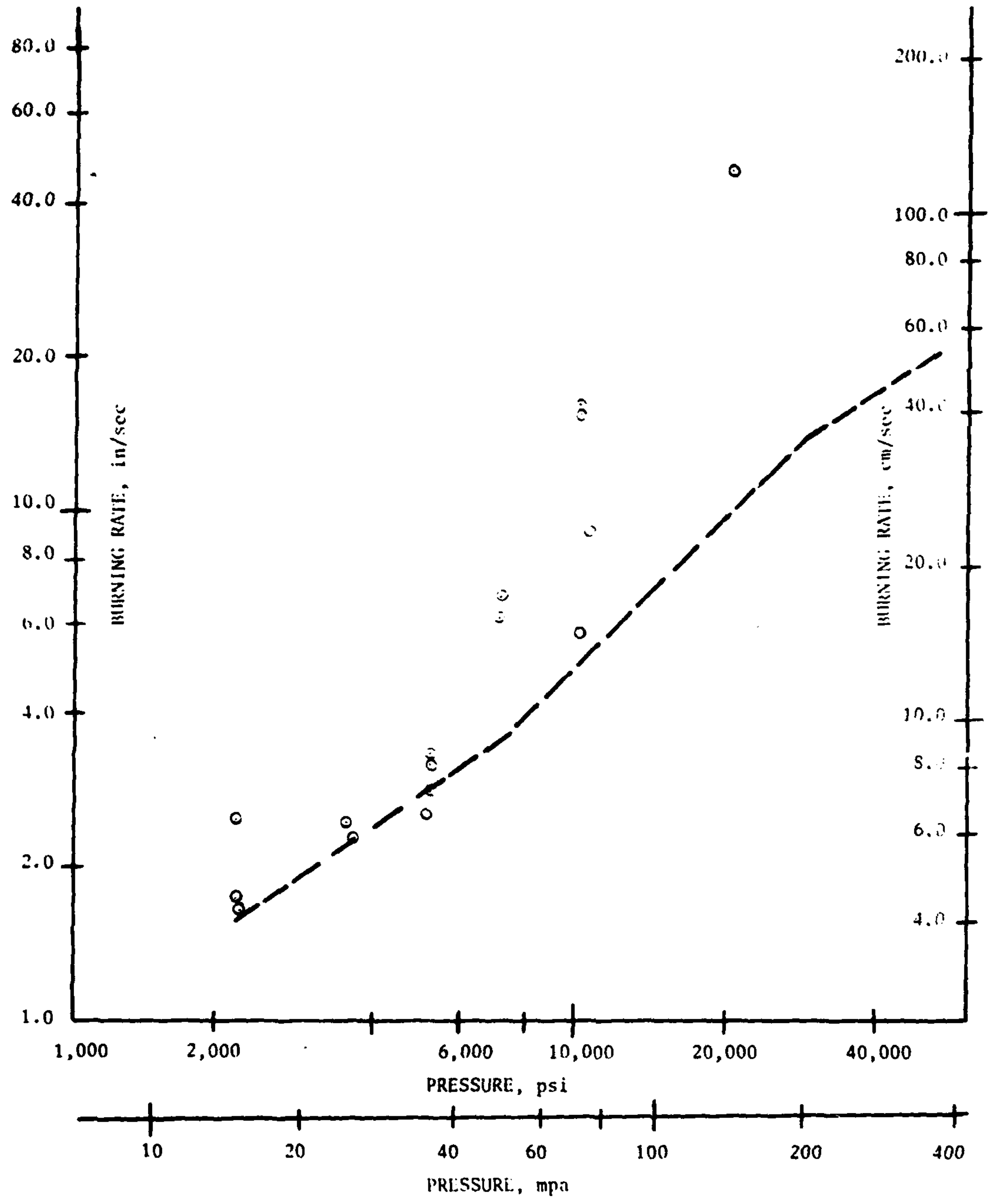

Figure 3. GP 172 Lot 1974, Ground

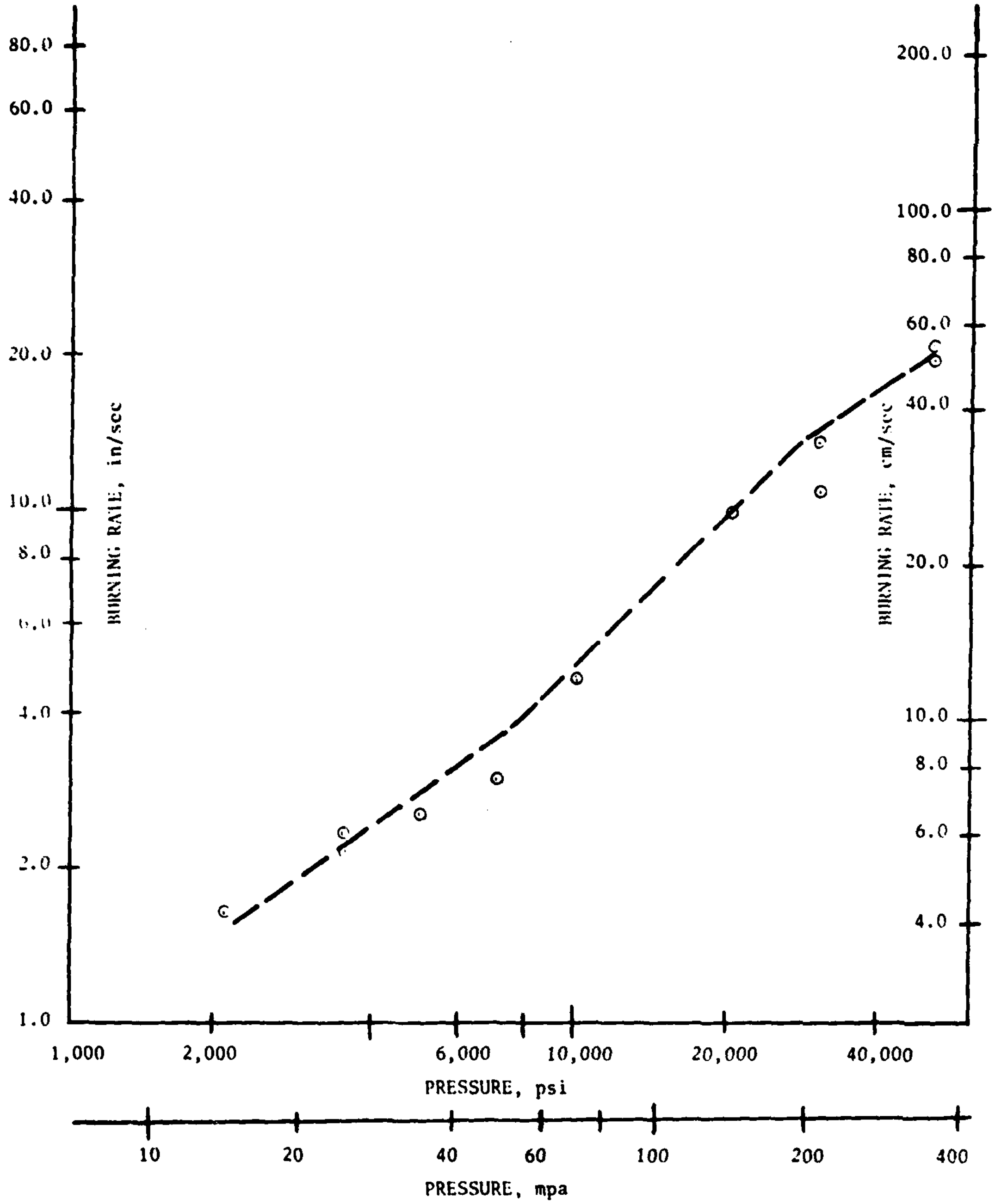

Figure 4. GP 200 Lot 1974 Recrystallized, Ground

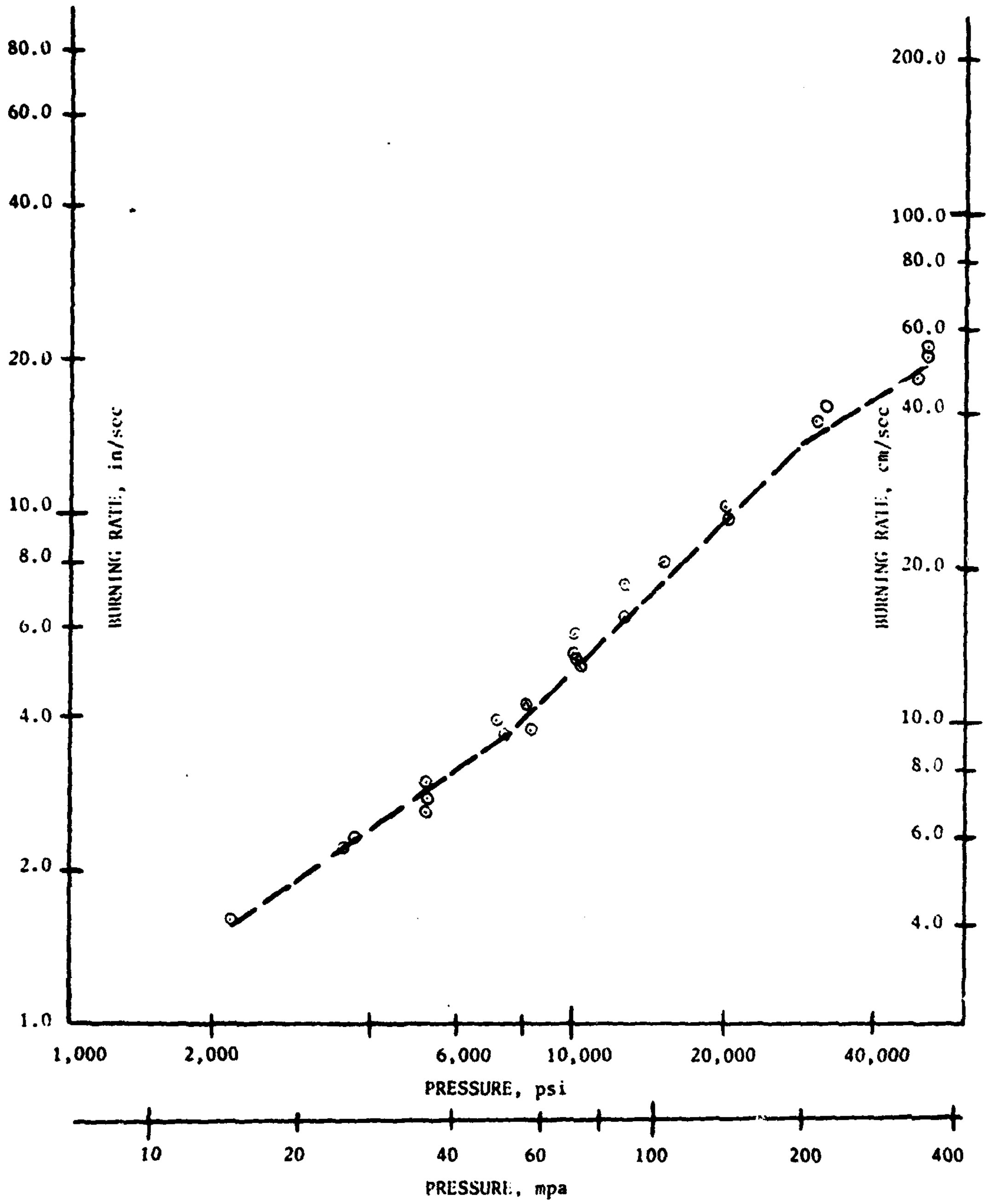

Figure 5. GP 218 R-4 Ground, IPA Wash

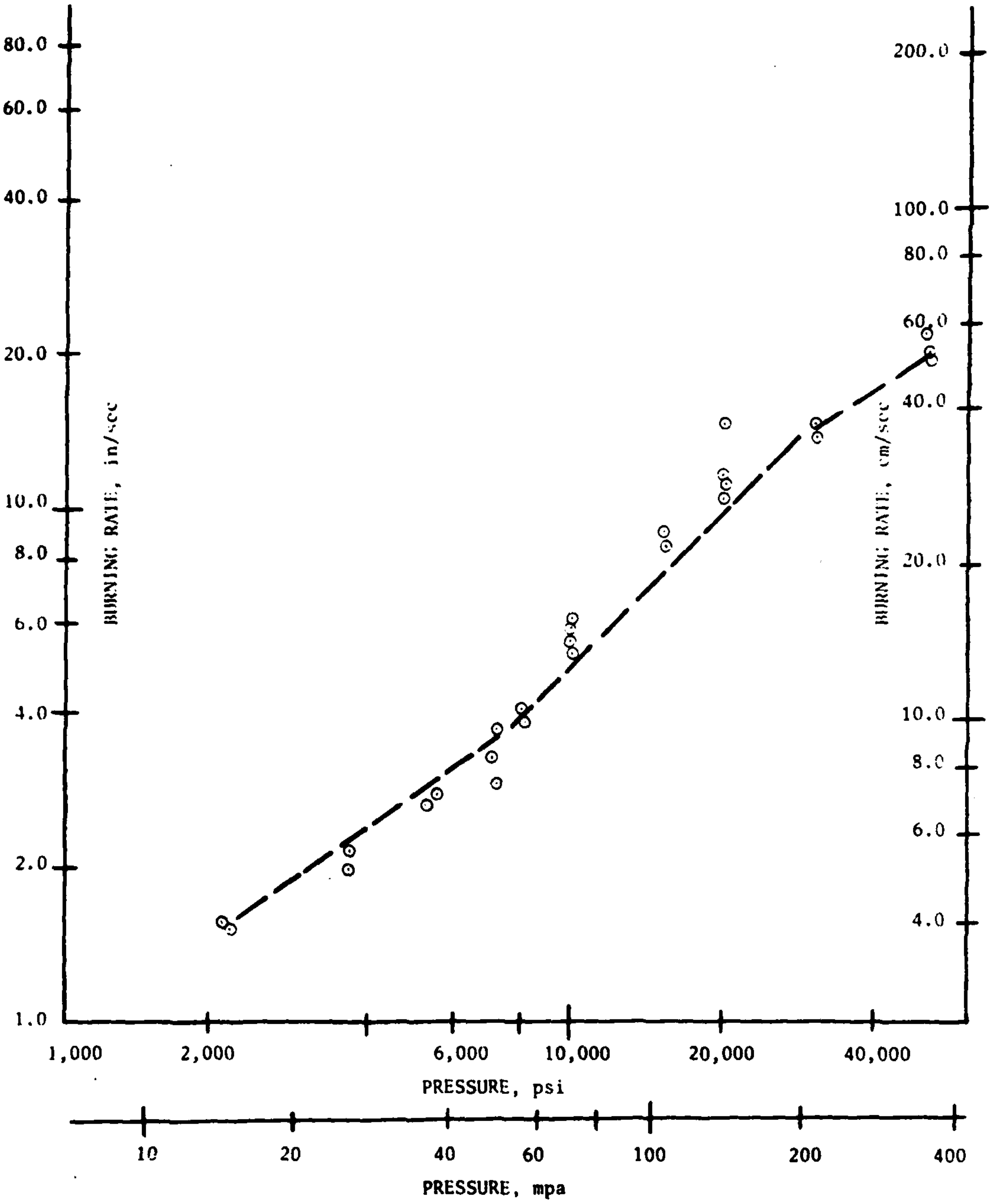

Figure 6. GP 219 R-4 Ground, IPA/Hexane, Hexane Wash

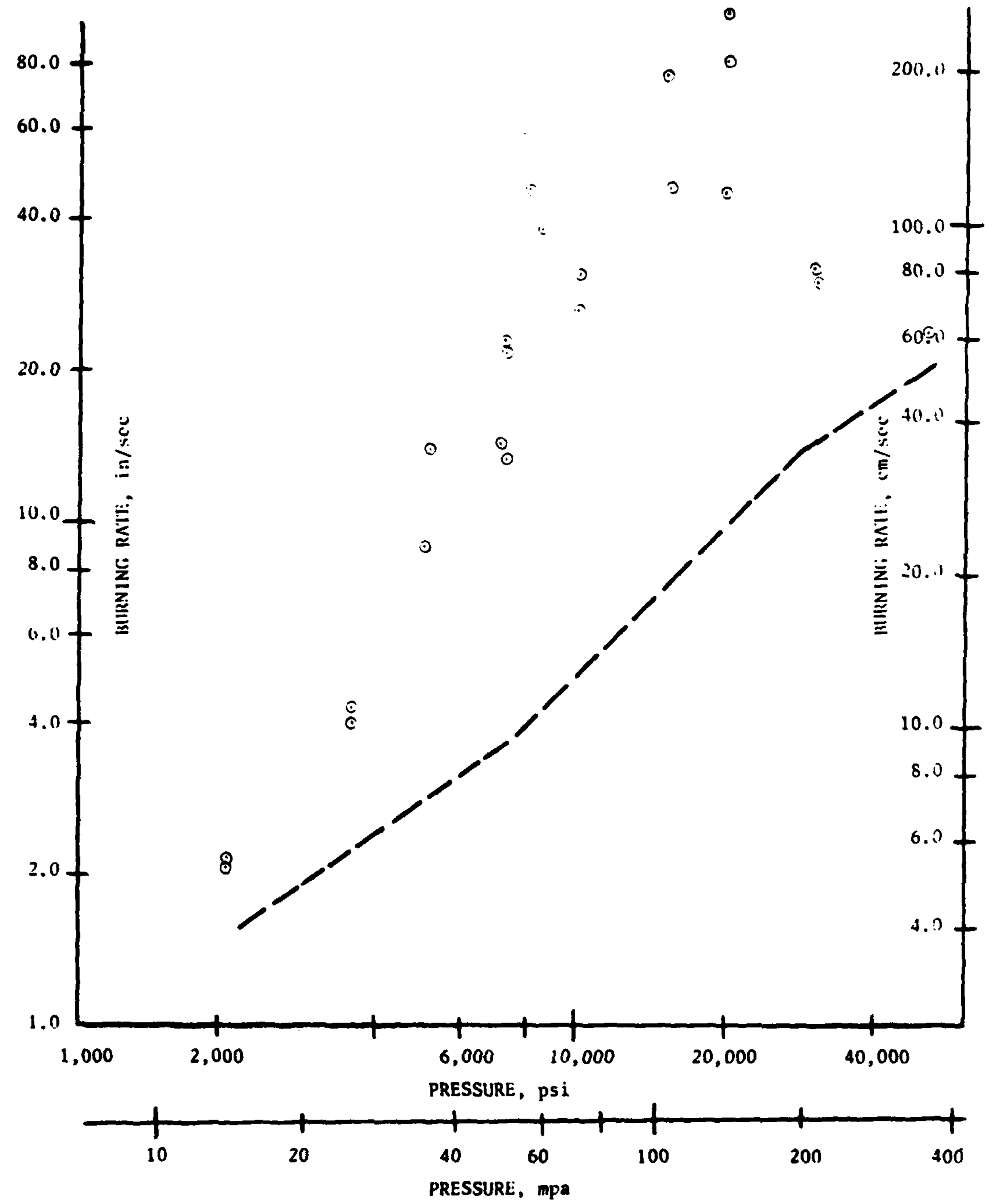

Figure 7. GP 228 Lot B-5, Ground, IPA, IPA/Hexane, Hexane, Freon® Wash

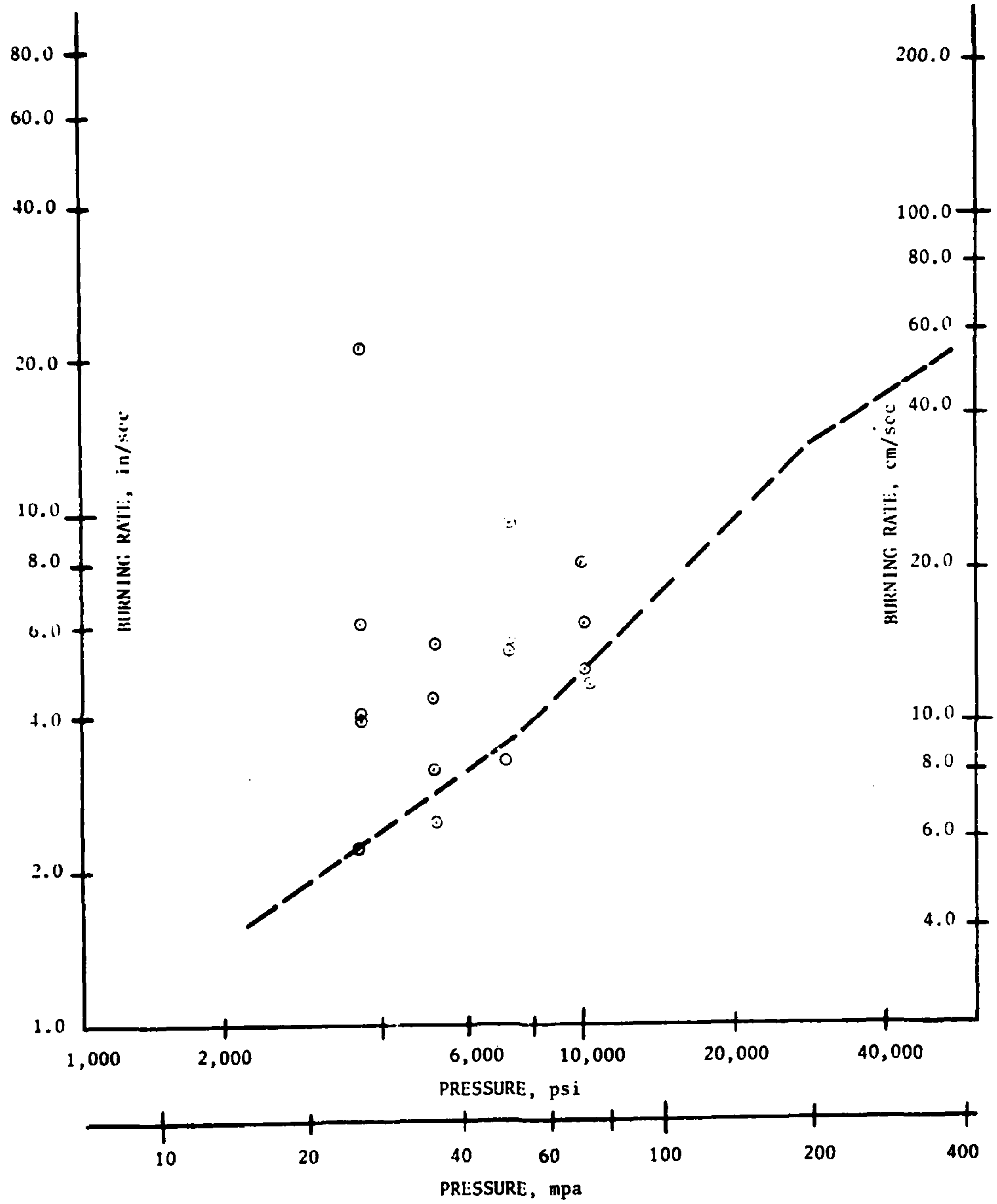

Figure 8. GP 234 Lot 2-7-1, Ground in Freon® 113

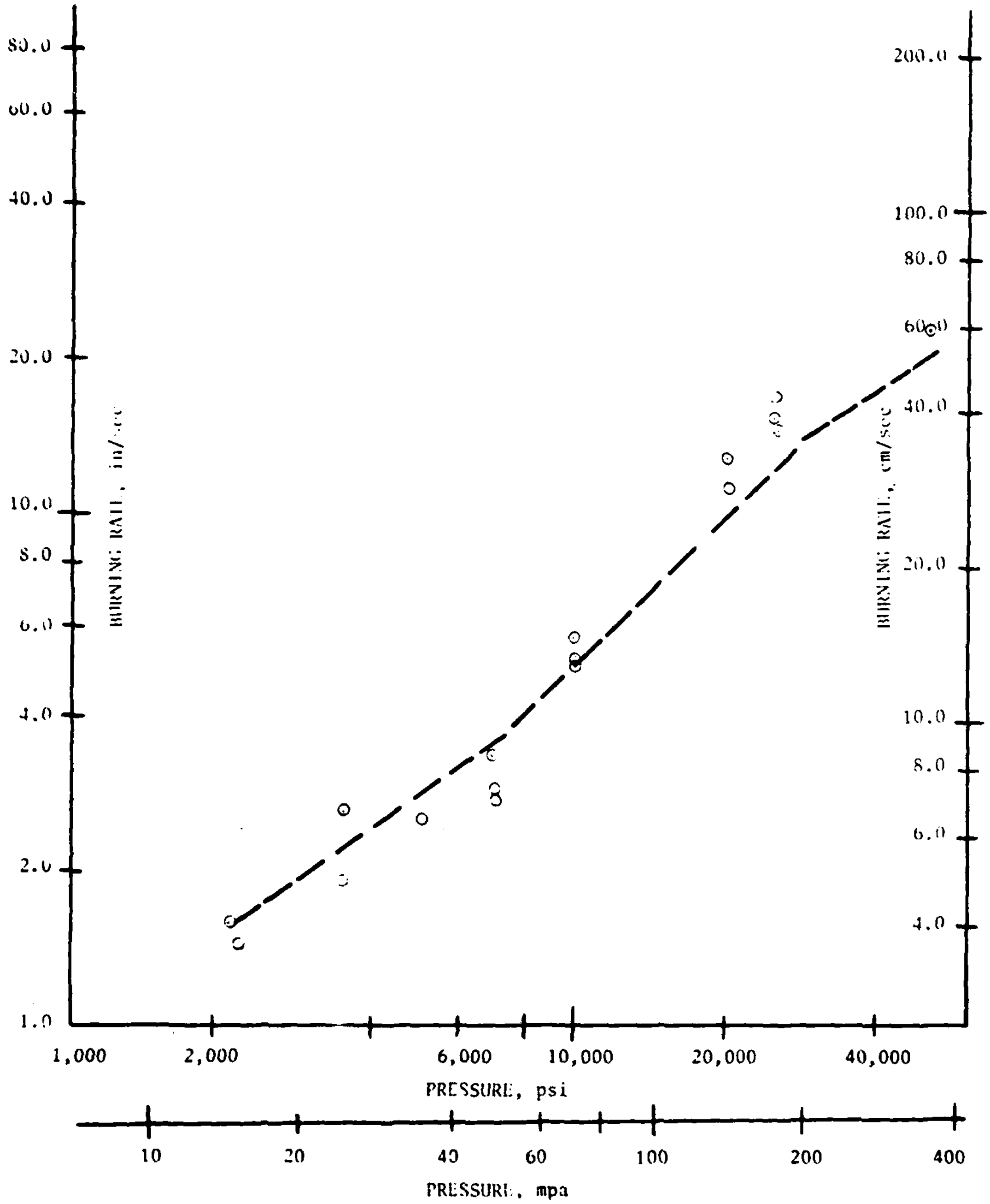

Figure 9. GP 246 Lot 2-7-1, Ground in Freon® 113, Air Oxidation

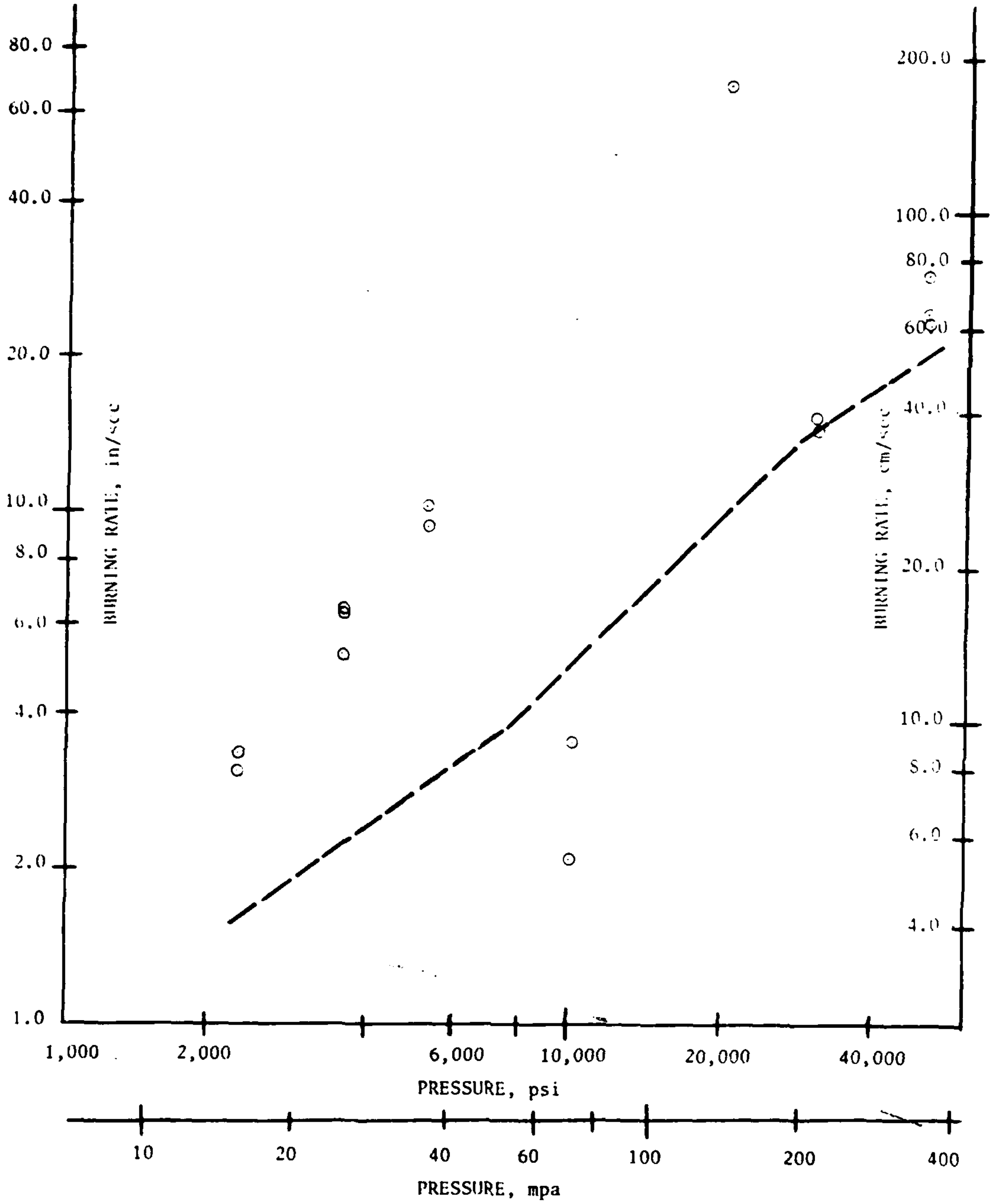

Figure 10. GP 247 Lot 2-7-1, Ground in Freon®, IPA Wash

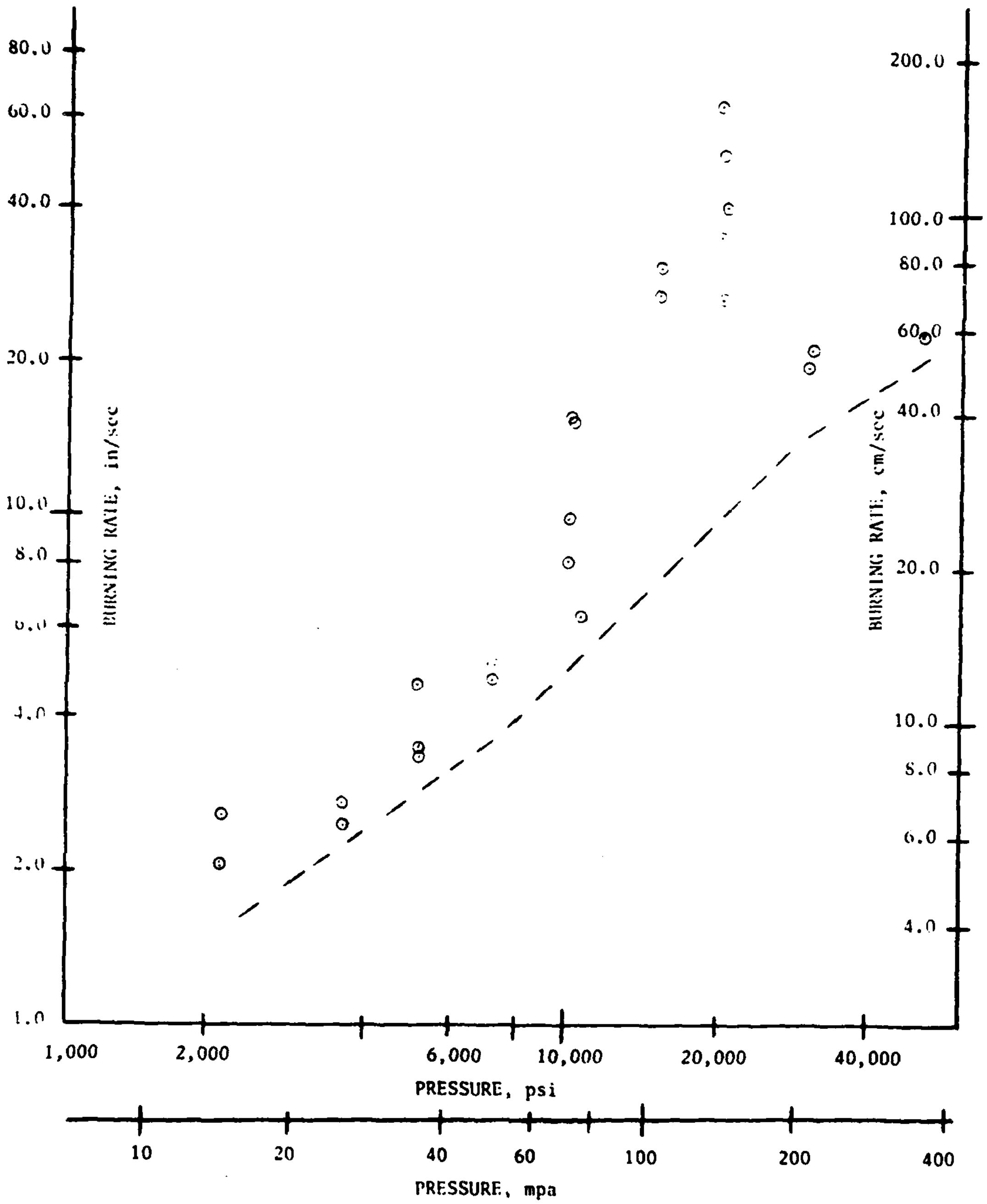

Figure 11. GP 248 Lot 2-7-1, Ground in Freon®, CH_2Cl_2 Wash

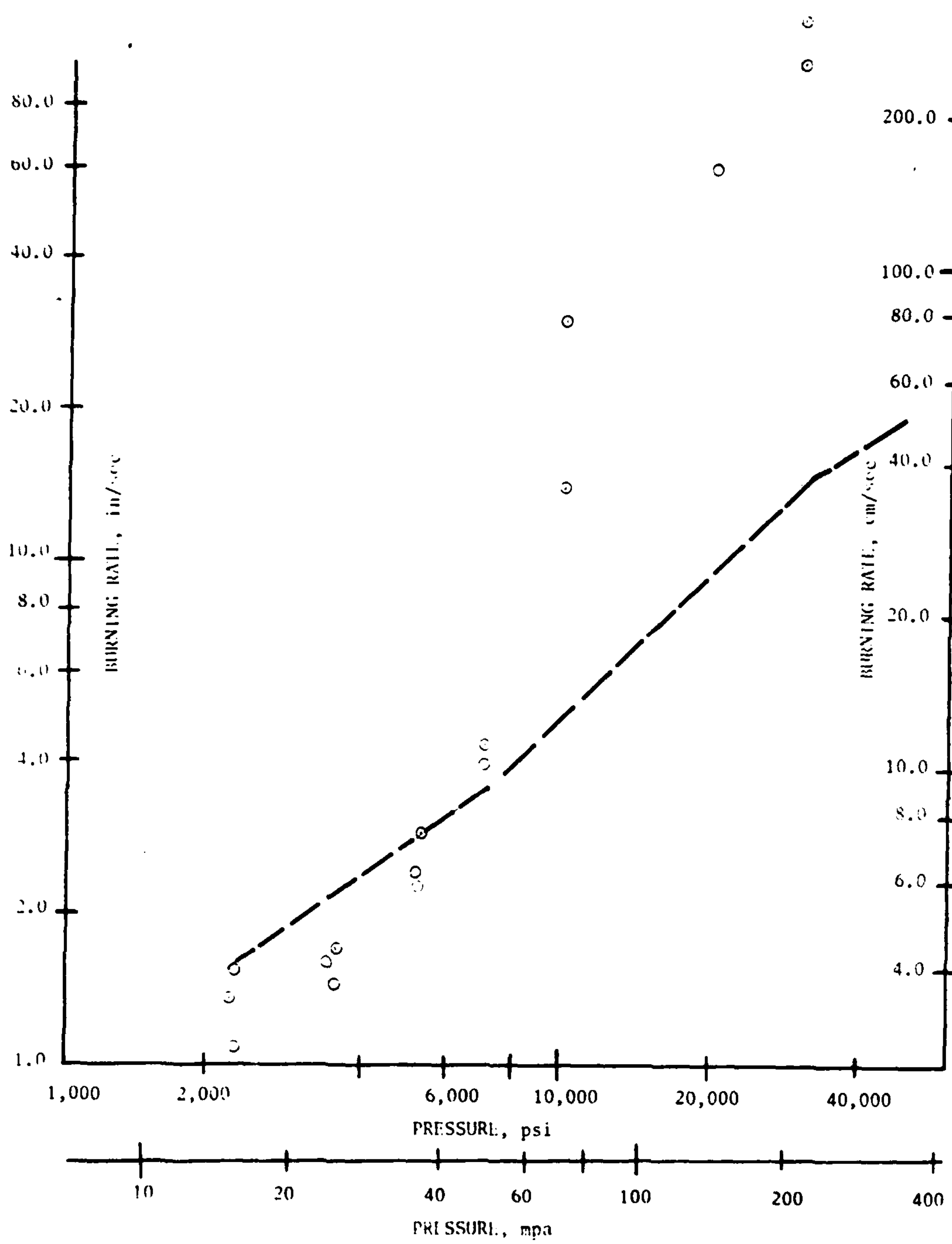

Figure 12. GP 215 Lot 2-7-1, Unground

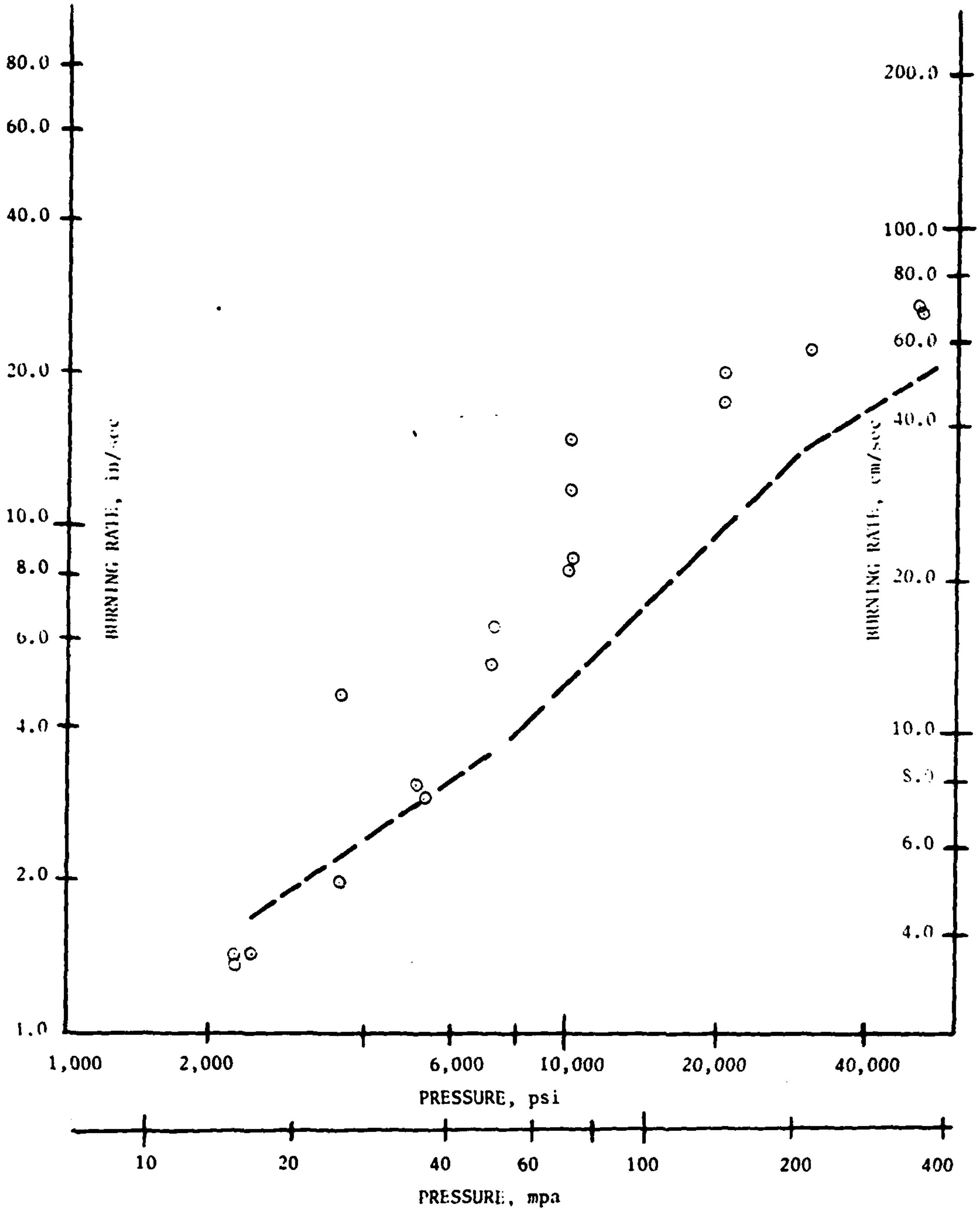

Figure 13. GP 215A Lot 2-7-1, Unground, Air Oxidation

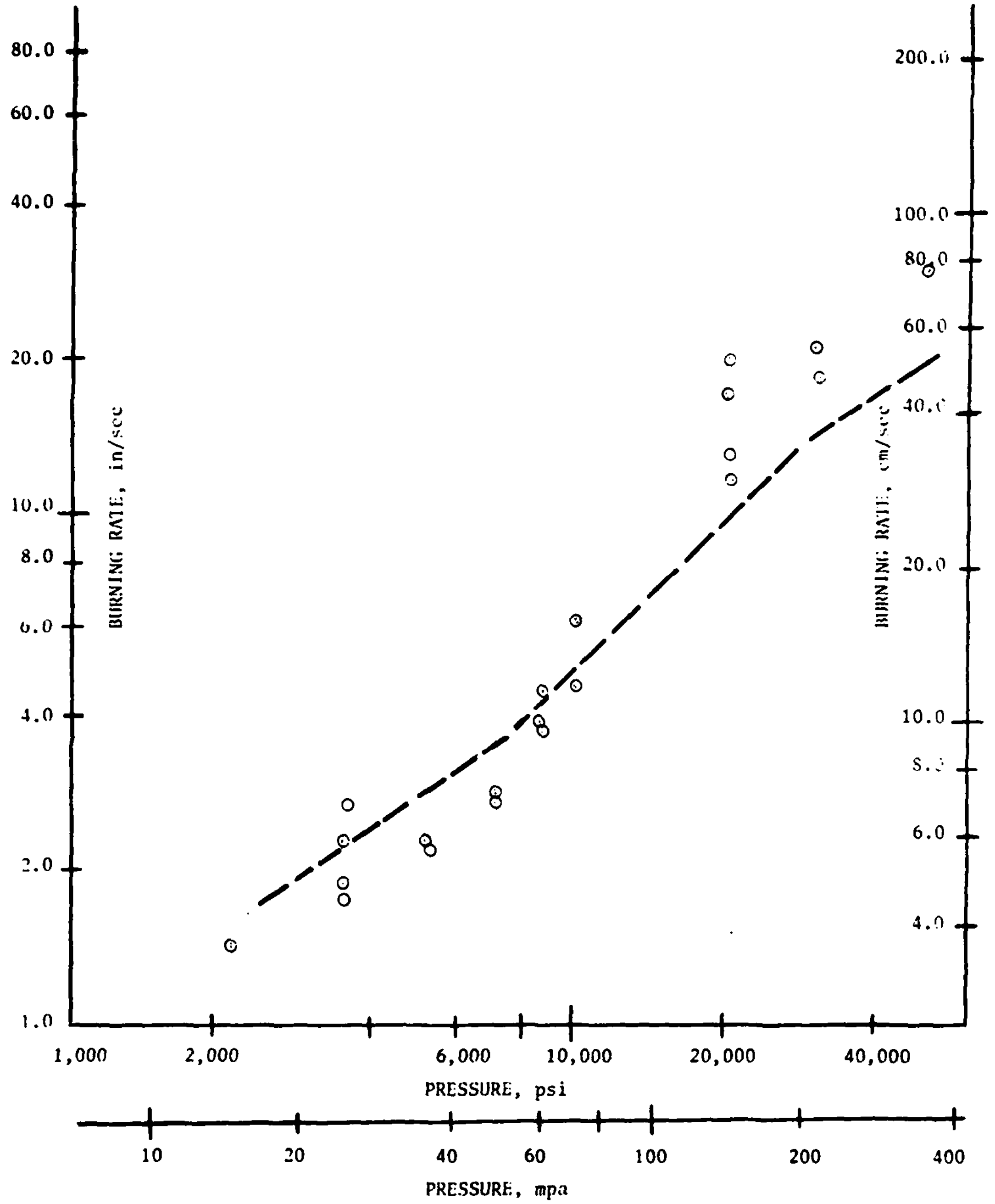

Figure 14. GP 216 Lot 2-7-1, Unground, IPA/Hexane, Hexane Wash

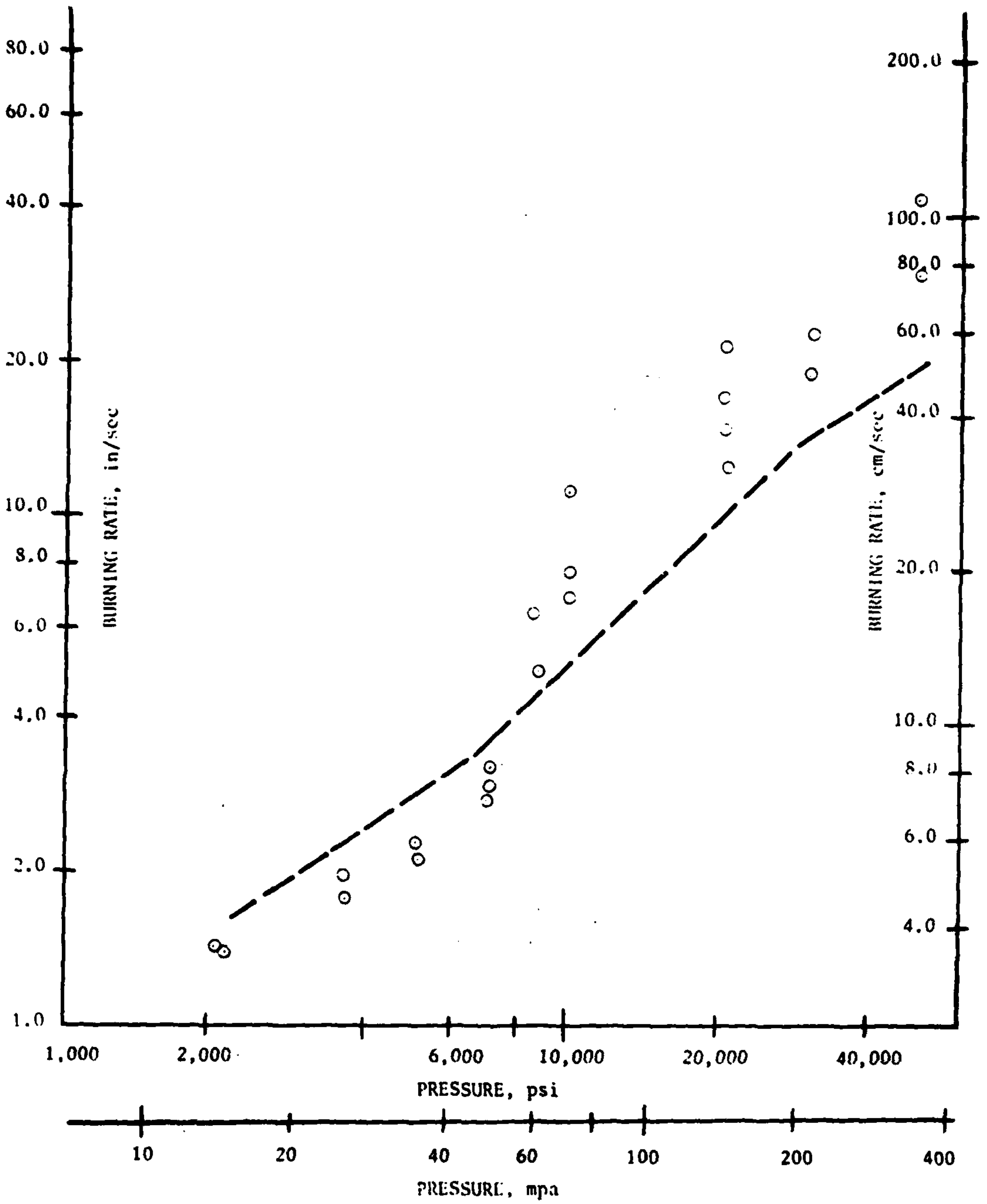

Figure 15. GP 217 Lot 2-7-1, Unground, IPA Wash

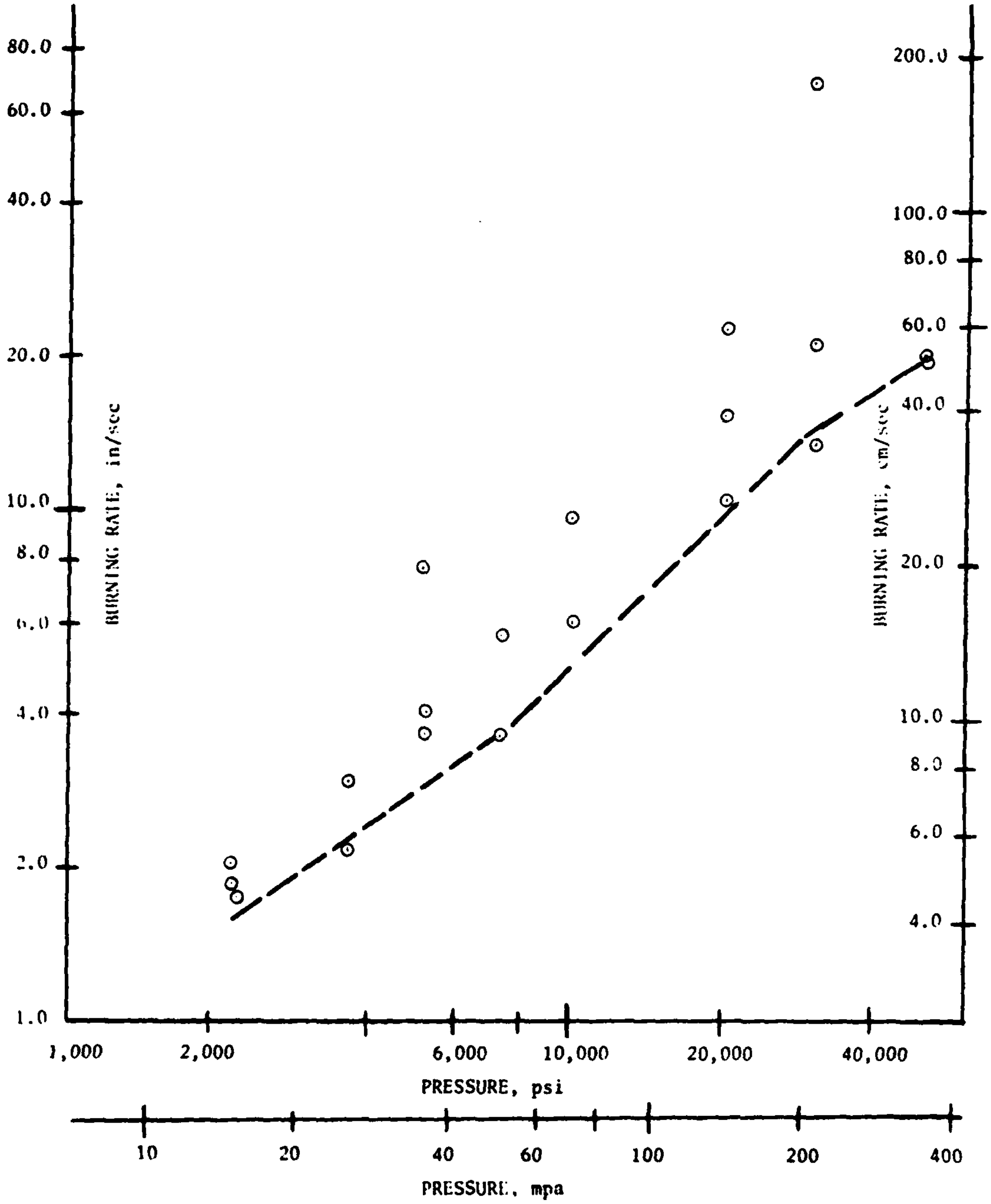

Figure 16. CF-1 Lot 2-7-1, Recrystallized, Ground

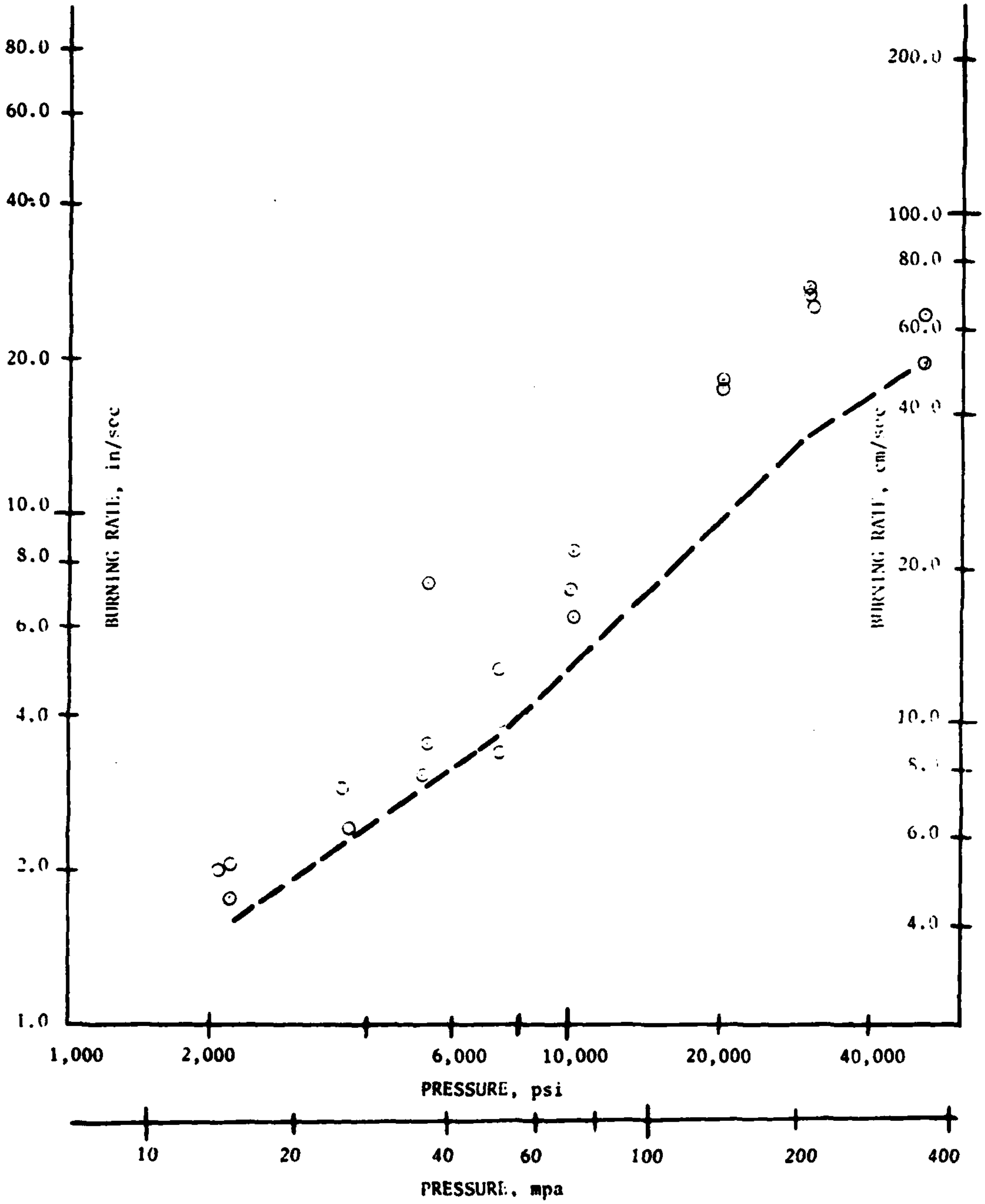

Figure 17. CF-2 Lot 6229, Ground

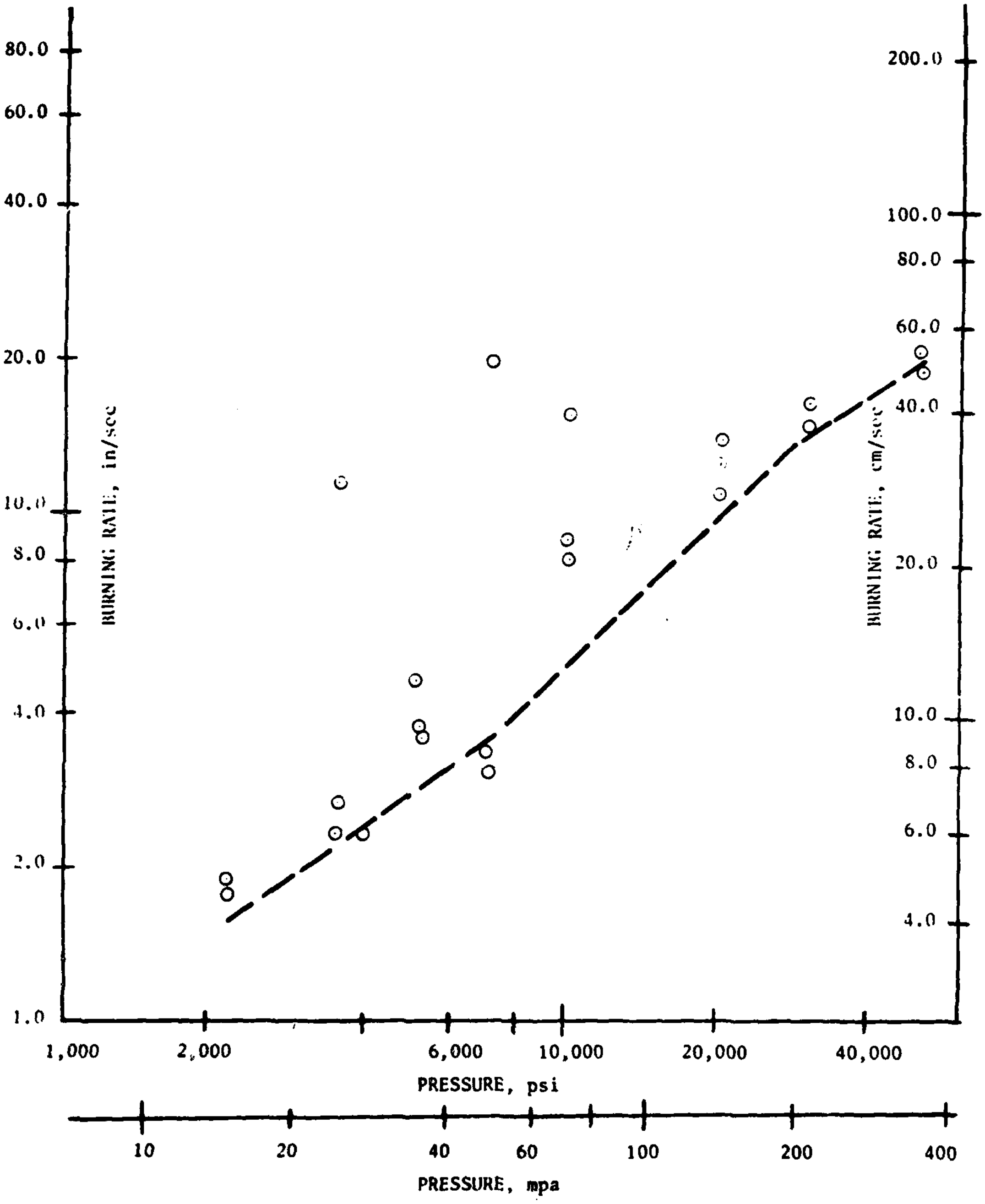

Figure 18. CF-3 Lot 2-7-5, Ground

APPENDIX A

TREATMENT OF TAGN

BATCH NO.	TAGN LOT NO.	TAGN TREATMENT
169	R-1	Ground* in ethyl acetate (ETAC), vacuum dried.
172	1974	Ground in ETAC, vacuum dried.
200	1974	Recrystallized three times, ground in isopropanol (IPA), air dried, designated as R-4.
218	R-4	Wicked with IPA, washed 3 times with IPA, air dried.
219	R-4	Wicked with IPA, washed 3 times with IPA, washed 3 times with IPA/hexane (50/50), washed 3 times with hexane, air dried.
228	B-5	Ground in ETAC, washed 3 times with IPA/hexane, washed 3 times with hexane, washed 3 times with Freon®, air dried.
234	2-7-1	Ground in Freon®, air dried.
246	2-7-1	Ground in Freon®, air dried with agitation.
247	2-7-1	Ground in Freon®, air dried, washed 3 times with IPA, air dried.
248	2-7-1	Ground in Freon®, air dried, washed 3 times with methylene chloride (CH_2Cl_2), air dried.
215	2-7-1	Air dried from container.
215A	2-7-1	Air dried with agitation.
216	2-7-1	Washed twice with IPA/hexane (50/50), 3 times with hexane, air dried.
217	2-7-1	Washed 3 times with IPA, air dried.
CF-1	2-7-1	Recrystallized twice, ground in IPA, air dried.
CF-2	6229	Ground in IPA, air dried.
CF-3	2-7-5	Ground in IPA, air dried.

*Grinding was accomplished in a 2.6-gallon SWECO Vibro-Energy Mill.

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